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(54) ASEPTIC SAMPLING

(71) We, SOCIETE DES PRODUITS NESTLE S.A., a Swiss body corporate, of Vevey, Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a device for aseptic sampling.

More particularly the invention relates to a device for aseptically taking samples of products held in a container, itself aseptic, in which they are being prepared, these products being more particularly for use in the industries concerned with the processing and manufacture of food products or the pharmaceutical industries.

Various types of devices for taking samples of products held in a container are already known. In these devices, the members in which the product circulates between the container in which it is held and/or is produced and the container for receiving it in the form of a sample are usually sterilised by means of high temperature dry steam. In certain devices, in addition to sterilisation by a steam barrier, certain parts are sterilised by chemical means such as, for example, a liquid disinfectant. With these various known devices it is necessary to provide and to operate increasing numbers of valves and circuits to increase sterility of sampling.. Moreover, when the sterility obtained with the steam is added to the sterility obtained by using a liquid disinfectant, the installations end up being quite complex and can only be operated by skilled personnel. On the other hand, if the devices are simple and easy to operate, only the installation remains sterile in practice while the sterility of the sample taken cannot be guaranteed.

The present invention obviates these disadvantages by providing a device for aseptic sampling, from a container which is aseptic itself, which, despite its simple structure guarantees the sterility of the samples taken and which is simple to operate owing to the fact that it may be operated by means of two valves only.

The present invention provides a device adapted to take samples of a product held in a container, itself aseptic, from the container, the device comprising a first pipe adapted to issue from the said container and ending at a sample receiving unit provided with a vessel for receiving products, which vessel may be joined in air-tight manner to the end of the said first pipe, a member for blocking the said first pipe, a second pipe for supplying a sterilising fluid at high temperature and high pressure which may be blocked by means of a blocking member and which opens into the first pipe at a point situated between the said blocking member and the sample receiving unit, and a third pipe issuing from the vessel receiving products at whose free end is arranged means for communicating with the atmosphere.

According to a preferred embodiment which ensures the sterility of the blocking member of the first pipe issuing from the aseptic container, the said first pipe comprises two branches, for example one horizontal and one vertical branch issuing from the horizontal branch. The two branches thus form a T whose horizontal branch which is adapted to issue directly from the container and which is blocked at its opposite free end, is provided with a needle valve the seat of which is adapted to be located between the outlet of the container and the inlet of the vertical branch and the needle body of which is supported by an airtight bearing and leaves an annular chamber round itself between the said bearing and its drive mechanism. The external wall of this annular chamber which is the external wall of the horizontal branch of the first pipe contains the means for placing the third pipe in communication with the atmosphere and this third pipe terminates in the annular chamber.

The pipe for supplying sterilising fluid

advantageously comprises a calibrated pipe branching round the blocking member thereof so as to allow for suitable regulation of the flow of sterilising fluid in to the installation during sampling.

The means for placing the end of the third pipe in communication with the atmosphere is advantageously formed by a calibrated orifice made through the wall of the first pipe at the level of the annular chamber surrounding the body of the needle of the needle valve.

The sterilising fluid is preferably dry steam at a pressure of at least 3 bar.

Other characteristics and advantages of the present invention will be shown better by the description given below as a non-limiting example with reference to the accompanying drawing in which the single figure is a diagrammatic view of the device for aseptic sampling according to the said invention.

With reference to the single figure: the device according to the invention for taking samples of products held in a container 1, which may equally well be a pipe or a tank, from this container, is formed by a pipe 2 connected to a unit 3 for receiving the said samples through a member 4 for opening and closing the pipe 2 interposed between said container and said unit for receiving the samples and by a member 5 for supplying aseptic gas into said pipe 2 and opening into said pipe 2 between unit 3 and member 4. In addition, the device comprises a pipe 6 communicating with the receiving unit and the atmosphere via a suitably calibrated evacuation member 7.

The pipe 2 is formed by a tube whose wall is designed, both with regard to the nature of the material used and to the thickness thereof, to resist, on the one hand, pressures of three bar or above, and, on the other hand, temperatures at least equal to that of the steam at the maximum pressures which it may reach in the pipe 2. For this purpose, the components of this pipe are preferably made of non-corrodible, high strength metal and the various components are argon welded to withstand high temperatures.

The pipe 2 is advantageously formed in two parts, a first part 2a having a longitudinal axis which is normal to the external surface of the container 1, generally horizontal, and a second part 2b, the axis of which is perpendicular to the longitudinal axis of the pipe position forming the first part, therefore generally vertical and branched to the first part at an intermediate point between the two ends thereof such that the assembly of these two parts 2a and 2b has the general shape of a T, the end of whose vertical branch terminates at the unit for receiving or sampling products emanating from the container 1. The horizontal part 2a of the pipe which is blocked at the end opposite the said container may be blocked at an intermediate point between the outlet of this container 1 and the inlet of the vertical part 2b. This effect is advantageously obtained by means of a needle valve of any suitable known type, the needle 4a of which rests on the seat 4b in the blocked position. The seat 4b rests on a shoulder of the pipe 2a situated between the container 1 and the point where the pipe 2b joins said pipe 2a and is sealed by means of any suitable gasket which resists high temperatures and which may advantageously be, for example, a seat made of Teflon ("Teflon" is a Registered Trade Mark), which is highly resistant to crushing. The needle 4a itself is supported by a bearing 4c machined into the part 4b and made airtight by means of at least one suitable gasket, preferably made of Viton ("Viton is a Registered Trade Mark), such that an annular chamber 8 remains, on the one hand, between said sealed bearing and the mechanism 4d for the longitudinal advance or return of the needle caused by the milled button 4e on the free end of the part 2a and on the exterior and, on the other hand, between the body of the needle and the internal lateral wall of the pipe 2a. The terminal extremity of the pipe 6 ends in this annular chamber 8. The wall of the pipe 2 contains an orifice 7 for communication with the atmosphere which forms the adjustable member for placing the pipe 6 in communication with the atmosphere via the annular chamber 8. The orifice 7 may be free and of a predetermined diameter suitably selected for obtaining the desired delivery rate. It may also be blocked, for example by means of a valve which is adjusted or adjustable to any exhaust pressure of suitably selected predetermined value.

The pipe 5 ends at the vertical part 2b of the pipe 2 between the end joined to the horizontal part 2a and the unit 3 for receiving the samples. The pipe 5 is connected to a source for producing an aseptic fluid such as, for example and preferably, dry steam at relatively high pressure, for example at at least 3 bar, therefore also at high temperature. A valve 9 of any suitable known type for controlling the intake of steam in the pipe 2 is interposed between this source for producing steam and the point where the pipe 5 ends at the pipe 2b. This valve is looped by a suitably calibrated branch or "by-pass" 10 which is formed by a pipe of small diameter which may be blocked by means of a valve 11, said diameter being determined so that it is possible to pass a flow of steam at a reduced rate when the valve in the pipe 5 is closed and this pipe consequently blocked.

The unit 3 for receiving samples which is located at the end of the vertical pipe 2b comprises a pipe element, one end of which is connected to the end of this same vertical pipe and the other end of which is connected to a screw threaded head 13. Screw-headed thread 13 is provided with a seal 14 made of any suitable known material, that is to say material which is resistent to crushing and to high temperatures but preferably made of Teflon for allowing tight screwing of an air-tight bottle or flask 15 having a screw-threaded neck, the seal being permanently installed in this very head 13 provided for this purpose. The seal 14 contains an orifice 16 so that the vertical pipe 2b communicates with the interior of the receiving bottle as well as an orifice 17 through which the interior of said bottle also communicates with the interior of the pipe 6 and, via the latter, with the annular chamber 8 and consequently with the atmosphere. The device just described allows a sterile sample to be taken aseptically from an installation functioning aseptically, that is to say from the container 1, while still maintaining sterility both in the installation and in the sample. The following mode of operation is adopted for this purpose.

In a first phase forming the phase preparatory the actual sampling operation, the needle valve 4 blocks the pipe 2a with its needle resting for this purpose on the seat 4b. The receiving bottle is screwed with its screw-threaded neck to the unit for receiving samples and the steam is introduced into the pipe 2b, the valve 9 being open for this purpose while the "by-pass" is blocked since the valve 11 is in the closed position. The steam thus admitted into the pipe 2b spreads on both sides of the orifice connecting the pipe 5 to the pipe 2b and, in particular, bathes the annular chamber surrounding the needle 4a in the pipe 2a and contained between the seat 4b and the bearing 4c. On the other side of this orifice, it travels at a very reduced rate along the circuit formed by the lower part of the pipe 2b from the interior of the receiving bottle 15, the pipe 6 and the annular chamber 8 surrounding the body of the needle valve between the bearing 4c and its drive mechanism 4d up to the means 7 for communication with the atmosphere. An almost constant pressure is maintained in the entire circuit described above owing to the adjusted means 7, or formed more simply by an orifice of small diameter of, for example, about 1 mm thus acting as a diaphragm (setting the effective aperture size) for the very slow delivery of the steam. The temperature and the pressure of the steam residing in the parts of the pipe shown are such that all parts are in a sterilising atmosphere. Consequently, when the needle valve is in the closed position and the steam inlet valve 9 in the open position, a steam barrier is formed and keeps the sample delivery pipe, the receiving flask and the rear of the toroidal seal of the said needle valve sterile.

This state of affairs, which may either be maintained permanently so as to allow sampling at any moment or may be brought about only a short while before sampling, precedes the latter whatever the circumstances.

Sampling by means of the device just described is carried out in the following manner. The flow of steam circulating in the circuit is firstly reduced to a mere thin stream either by keeping the by-pass 10 closed and the valve 9 ajar or preferably by keeping said valve 9 closed and by opening the valve 11 of the calibrated by-pass. The operator wears thermally insulating gloves made, for example, of asbestos, and in a first stage rapidly sets down the receiving bottle 15, the neck of which is kept in the vicinity of the flame of a gas burner. (This gas burner may be portable, for example of the Bunsen type, but must then preferably be held by a second operator, or may be installed permanently and lit by the operator by means of a pedal control.) Then, in the second stage, the operator replaces this receiving bottle with an identical bottle which has previously been sterilised in a laboratory by passing a flame through the orifice as in a normal microbiology operation and which is screwed on the seal 13. Once this sterile bottle has been installed in in an air-tight manner, it is filled by merely opening the needle valve 4 causing the container 1 to communicate with the pipe 2 and the sampling bottle. The product flows into the sampling bottle (which is at atmospheric pressure) because the pressure in container 1 is higher due to the high temperature (sterile conditions) in the container. The orifice which has been closed up until now is opened by the needle of the said valve, and thus causes a proportion of the solid or liquid product held in said container to pass from said container into the bottle. The circulation of the said product between its point of origin and its target takes place in a pipe which has previously been made aseptic and entirely sterilised by a steam barrier in the manner described above. The small stream of steam injected through the pipe 5 prevents a proportion of the sampled product from passing in this same pipe. It is obvious that the flow-rate of the sampled product may be varied, and with it the rate of sampling, by opening the needle of the valve 4 to a greater or lesser extent. Once the desired quantity of products has been received in the sampling bottle, the needle valve 4 is closed thus again blocking the horizontal part 2a of the

pipe and consequently preventing the product emanating from the container 1 from entering this same pipe. Once the bottle has collected the sample, it is removed in the same manner as the previous receiving bottle, then the previously sterilised receiving bottle is replaced.

During sampling as described above, a little steam condenses in the sampled product. In the majority of cases, particularly when the sampled product is liquid, this slight addition does not obstruct the series of analytical operations.

In particular cases of analysis of dry extract, the tests carried out showed that sampling could take place without injecting a small stream of steam into the circuit during this same sampling process without impairing sterility. This is explained by the fact that when the sampling bottle was filled, the sterile air which it contained is flushed away from it in the pipe while the diaphragm provided for evacuating the steam keeps this zone under sterile superpressure.

WHAT WE CLAIM IS:—

1. A device adapted to take samples of a product held in a container, itself aseptic, from the container, the device comprising a first pipe adapted to issue from the said container and ending at a sample receiving unit provided with a vessel for receiving products, which vessel may be joined in airtight manner to the end of the said first pipe, a member for blocking the said first pipe, a second pipe for supplying a sterilising fluid at high temperature and high pressure which may be blocked by means of a blocking member and which opens into the first pipe at a point situated between the said blocking member and the sample receiving unit, and a third pipe issuing from the vessel for receiving products at whose free end is arranged means for communication with the atmosphere.

2. A device for aseptic sampling as claimed in claim 1, wherein the first pipe which is adapted to issue from the aseptic container consists of two branches, a horizontal branch and a vertical branch issuing from the horizontal branch which thus form a T, the horizontal branch which is adapted to issue directly from the container, being blocked at its free opposite end and being provided with a needle valve whose seat is adapted to be located between the outlet of the container and the inlet of the vertical branch and whose needle body is supported by a sealed bearing leaving an annular chamber round it between the said bearing and the drive mechanism of the needle body, the external wall of the said annular chamber, which is the external wall of the horizontal branch of the first pipe, containing the means for placing the third pipe in communication with the atmosphere and the third pipe terminating in the said annular chamber.

3. A device for aseptic sampling as claimed in Claim 1 or 2, wherein the pipe for supplying sterilising fluid includes a calibrated pipe branching round the blocking member thereof.

4. A device for aseptic sampling as claimed in Claim 2, wherein means for placing the end of the third pipe in communication with the atmosphere is formed by a calibrated orifice made through the wall of the first pipe level with the annular chamber surrounding the body of the needle of the needle valve.

5. A device for aseptic sampling according to any of Claims 1 to 4, wherein the sterilising fluid is dry steam at a pressure of at least 3 bar.

6. A device for aseptic sampling substantially as described with particular reference to the accompanying drawing.

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1 SHEET This drawing is a reproduction of the Original on a reduced scale